
**Rubber — Identification of accelerators
in cured and uncured compounds**

*Caoutchouc — Identification des accélérateurs dans les mélanges
vulcanisés ou non*



Foreword

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International Standard ISO 10398 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

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Printed in Switzerland

Rubber — Identification of accelerators in cured and uncured compounds

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1 Scope

1.1 This International Standard specifies methods using gas chromatography (GC) and thin layer chromatography (TLC) for the separation and identification of the following classes of accelerators in vulcanized and unvulcanized compounds:

- thiazoles
- sulfenamides
- thiurams and dithiocarbamates
- guanidines
- dithiodimorpholine

1.2 When 2-mercaptobenzothiazole (MBT) is identified and no sulfenamides are present, it is not possible to establish if the original accelerator was MBT and/or its salts or 2,2'-dibenzothiazoledisulfide (MBTS) as each of these accelerators may be produced from the others during the vulcanization process.

1.3 When sulfenamides are identified, it is not possible to establish if MBT and/or its salts and MBTS are present as these accelerators may be produced from 2-mercaptobenzothiazole sulfenamides during the vulcanization process.

1.4 The methods do not distinguish thiurams and dithiocarbamates derived from the same amines.

1.5 From the morpholine identification it is not possible to determine if the initial accelerator is 2-morpholiniothio-benzothiazole (MBS) or dithiomorpholine as morpholine may be formed from MBS and dithiodimorpholine during the vulcanization process.

1.6 The separation of accelerator compounds from unvulcanized compounds is relatively straightforward whereas separation from vulcanized compounds is difficult due to the lesser ability of solvents to penetrate the vulcanized matrix.

1.7 Some compounding ingredients may interfere with method B. In such cases methods A or C shall be used.

2 Principle

2.1 Method A — Identification of amines via GC and thiazoles via TLC

2.1.1 A portion of the sample compound is refluxed with hydrochloric acid (HCl) to hydrolyze sulfenamides, thiurams and dithiocarbamates. The resulting amine hydrochlorides are separated and purified. After purification, the amines are separated by GC as their trifluoroacetamide derivatives. Identification may also be effected by comparison of GC retention times of sample and standard trifluoroacetamides, prepared and analyzed under the same analysis conditions.